

1ST AND 2ND ORDERS		PROCESSES AND PROPERTIES INDEX		14D AND 4TH CROSS	
<div style="display: flex; justify-content: space-between;"> <div style="width: 40%;"> <p>FERLAN, F.</p> <p>4</p> </div> <div style="width: 50%; text-align: right;"> <p>28</p> </div> </div>		<p>The hydrolysis of rice hulls. Maka Samec, F. Ferlan, and A. Pajk (Chem. Studies Acad. Sci. and Arts, Ljubljana, Yugoslavia). <i>Abstr. Znanosti Umjetnosti Ljubljani, Kem. Lab., Kem. Studije</i> 1947, 52-75 (English summary).</p> <p>The authors preserve the sugars by drying the rice hulls and grinding them before the extn. Dried rice hulls (100 g.) soaked in 85% H_2SO_4 until swollen and then hydrolyzed in 10% H_2SO_4 at 100° for 5 hrs. yielded 48 g. of reducing sugars (60% of the org. matter) which had an agreeable taste, the smell of caramel, gave osazones in 187-197%. At 5° 1 part of dried rice hulls hydrolyzed in 10 parts of 38% HCl for 66 hrs. yielded 0.30 part of a reducing sugar (37% of the org. substance); the acid ext. was black, and even when the acid had been removed, portions of the sugar carbonized while the ext. was being concentrated. Two parts of rice hulls extd. in 10 parts of 0.0-1.0% H_2SO_4 at 150-190° for 30 min. yielded 0.76 part of a reducing sugar (54.7% of the org. substance) which was fermented by brewers' yeast. During the first half of the extn. the yield consisted of pentosans; later it consisted of hexoses principally. When distd. with HCl these sugars yielded furfural. Frank Mareš</p>			
<p>ASAC 514 METALLURGICAL LITERATURE CLASSIFICATION</p>					
<p>1ST AND 2ND ORDERS</p>		<p>PROCESSES AND PROPERTIES INDEX</p>		<p>14D AND 4TH CROSS</p>	

FERLAN, M

YUGO .

Synthesis of 2,4-dinitrophenyl 2-quinolyl ether. M. Ferlan (Tvrzica dubika, Ruse, Yugoslav.). *Vestnik Sloven. hem. drustva* 1, 9-10 (1954).—A mixt. contg. 48.3 g. 2-hydroxyquinoline, 67.3 g. 2,4-(O₂N)₂C₆H₃Cl, 174 cc. of 90% EtOH, and 18.7 g. KOH in 20 cc. H₂O, was refluxed 3 hrs. at 80° with const. stirring; when the mixt. had cooled, the ppt. was filtered, washed with 250 cc. cold 90% EtOH, suspended in 200 cc. warm H₂O, boiled with a small amt. of C, 70 cc. concd. HCl added, and the mixt. filtered hot; the filtrate yielded golden-red hexagonal crystals, which were filtered, washed with EtOH, suspended in 200 cc. H₂O, and treated with a concd. soln. of Na₂CO₃ until alk. to phenolphthalein, then with a few drops of 2N NaOH, and the pptd. ether washed with cold H₂O until free of Na⁺, yield, 72%, m.p. 164°.

N. Plavšić

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FERLAN, M.

YUGOSLAVIA/Chemical Technology. Chemical Products and Their
Application. Ceramics. Glass. Binding Materials.
Concrete.

H

Abs Jour: Ref Zhur-Khim., No 10, 1959, 35664.

Author : Dokszezanin, S., Ferlan, M., Kotnik, S., Lindtner, M.,
Ozim, V., Sonnenwald, S.

Inst : Slovene Chemical Society

Title : Yugoslav Corundum Production.

Orig Pub: Vestnik Slov Kem Drustva, 4, No 1-2, 65-68 (1957) (in
Slovene with an English summary)

Abstract: Geological and chemical prospecting has led to
the discovery of a deposit of bauxites suitable for
the production of corundum. A plant constructed at
the site is now furnishing all of Yugoslavia's abra-
sives demand. -- From a summary by the authors.

Card : 1/1

H-39

FERLAN, Zoran, inž.; PAVLOVIC, Sonja, inž.

Complexometric determination of sulfates in water.
Rudar glasnik no.4:132-134 '62.

1. Rudarski institut, Biro za analitičku hemiju, Beograd
(for Pavlovic).
2. ZRMK, Ljubljana.

CA

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Lithium aluminium hydride and kindred agents in organic chemistry. Josef Rudinger, Miloslav Fexles, and Miroslav Protiva. *Chem. Listy* 45, 309-76 (1951).—An exhaustive review with 559 references (literature reviewed up to the end of June, 1951). M. Hudlický

Chemical Abstr.
Vol. 48 No. 6
Mar. 25, 1954
Organic Chemistry

Lithium aluminum hydride and kindred agents in organic chemistry. In: Miroslav Ferlic and Josef Rulinger (Tech. Univ. Prague), Chem. Listy 47, 91-163 (1953).—A supplement to an earlier review (C.A. 46, 4403h) involving literature from July 1951 to June 1952. The review contains 413 references and tables of all compounds reduced by LiAlH_4 -type agents. M. Hudlický

4. Methylquinuclidine, Rudolf Lukes and Miloslav Beres,
Prague, Czech. Chem. List. 1934, 29, 1133.
Methylquinuclidine has been prepared by
the reaction of 1-methyl-4-ethylpiperidine (I)
with HBr (II) which was trans-
formed into CH_3Br (III) and reacted with N-ethyl-
4-methylquinuclidine (IV). The reduction of I in bulk
with H_2 gave, in addition to II, $\text{CH}_3\text{CH}_2\text{OCH}_2\text{CH}_2\text{CMe}_2\text{CH}_2$
(V) which with HBr yielded $\text{CH}_3\text{CH}_2\text{OCH}_2\text{CH}_2$ -
 $\text{CH}_2\text{CMe}_2\text{CH}_2$ (VI) and III. The 2nd method of
preparation of V with $\text{CH}_3\text{CO NH CO CH}_2\text{CMe}_2\text{CH}_2$
which whose diester (VIII) with LiAlH_4 gave CH_3CH_2 -
 $\text{OCH}_2\text{CH}_2\text{CMe}_2\text{CH}_2\text{OH}$ (IX), whose transformation
with HBr gave $\text{CH}_3\text{CH}_2\text{NHCH}_2\text{CH}_2\text{CMe}_2\text{CH}_2$
which reaction with NH_3 yielded IV. The
reaction of V with LiAlH_4 gave $\text{CH}_3\text{CH}_2\text{OCH}_2\text{CH}_2$
 $\text{CH}_2\text{CMe}_2\text{CH}_2$ (X). XI Analogous series of reactions
with $\text{Me}_2\text{C}(\text{OCH}_2\text{CH}_2\text{CMe}_2\text{CH}_2\text{OH})$ (XII) led
to $\text{CH}_3\text{CH}_2\text{OCH}_2\text{CH}_2\text{CMe}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{CMe}_2\text{CH}_2$ (XIII)
and $\text{CH}_3\text{CH}_2\text{NMeCH}_2\text{CH}_2\text{CMe}_2\text{CH}_2\text{Br}$ (XIV), to the
quaternary of 1-methylquinuclidinium salts (XV). MeC-

[illegible]

Re. 10/10/10

212-

[illegible]

1. 1000 ft. m. 140 50'; Me ester with

CH_3N_2), m. 92-3° (from C_6H_6). $\text{MeC}(\text{CH}_3\text{CO}_2\text{H})_2$ (4 g.) dissolved in warm H_2O and distilled with 2 ml. 31% aq. Me_2NH ; gave distillate mp 94-5° (lit. 93-5°). XII

1990-1991

1. The first group of people who are not in the labor force are those who are not in the labor force because they are not in the labor force.

...the ...

$$\frac{1}{\sqrt{2\pi}} \int_{-\infty}^{\infty} e^{-\frac{1}{2}x^2} dx = 1$$

LUKES, R.; FERLES, M.

4-methylquinuclidine [in German with summary in Russian]. Sbor. Chekh.
khim. rab. 18 no. 6: 818-828 D '53. (MLRA 7:6)

1. Institut obshchey eksperimental'noy khimii Prazhskogo Khimicheskogo
instituta. (Quinuclidine)

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FERLES, Miloslav

/ Löffer-Freytag reaction. Rudolf Lukš and Miloslav
Ferles. Collection Czechoslov. Chem. Commun. 25, 1227-04 (1)
36 (1956) (in German).—See C.A. 49, 10210d. B. J. C.

Miloslav Perles

Contribution to the Lomer-Florey reaction. Rudolf Luket and Miloslav Perles (Vrška škola chem. technol. Prague). *Chem. Zvesti* 10: 810-18 (1955); cf. Liller and Florey, *C.A.* 4, 174. The action of H_2SO_4 and ultraviolet light upon *N*-chloro derivs. of 4-ethylpiperidine (I) and 4-methyl-4-ethylpiperidine (II) resulted in the formation of 7-methyl- (III) and 4,7-dimethyl-1-azabicyclo[1.2.2]heptane (IV). The 5-membered ring was thus formed preferentially to a 6-membered one. As a reference material,

III was synthesized starting with $O.CO.CH_2.CH.CMe.O.CO.CH_2$ (V) via $HOCMe(CH_2CH_2OH)_2$ (VI) and $BrC-$

$Me(CH_2CH_2Br)_2$ (VII). During the reduction of 4-(β -hydroxyethyl)pyridine (VIII) with Na in BuOH, I and 4-ethyl-1,3,5,6-tetrahydropyridine (IX) were obtained in addn. to the main product, 4-(β -hydroxyethyl)piperidine (X). I was prepd. by reduction of 4-ethylpyridine (XI) with Na in BuOH, IX being a by-product. Adding 181 g. Na to a heated soln. of 58.5 g. VIII in 250 ml. BuOH, refluxing the mixt. 7 hrs., treating with H_2O , sepg. the BuOH layer, removing the BuOH by steam distn. after acidifying with HCl, mixing the residue after the steam distn. with the original alk. aq. layer, extg. the bases with Et_2O , and distg. the ext. gave 5.5 g. IX, b_m 157-8°, and 22.5 g. X, b 136-41°. The ether distillate contained 0.1 mole of bases from which were prepd. picrates: 15 g. of a picrate of IX, m 160-2° (from EtOH); IX, b_m 165°, and 1 g. of a picrate of I, m 162-3° (from EtOH). Similar reduction of 188.5 g. XI in 400 g. BuOH with 485 g. Na gave 87 g. I, b_m 150-4° (aryl deriv. m , 78-9° (from petr. ether-benzene)), and 40 g. IX, b_m 157-82°. X was transformed to quinuclidine [picrate, m 255° (from aq. EtOH), methiodide m 343° (from EtOH-AcOEt)]. Adding during 1 hr. a soln. of 10 g. V in 100 ml.

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Rudolf L. L. L.

tetrahydrofuran to a mixt. of 4.5 g. LiAlH₄ in 100 ml. of the same solvent, refluxing the mixt. 2 hrs., decanting, with 100 ml. 10% H₂SO₄, etc. the ppt. and boiling with 100 ml. 10% H₂SO₄, etc. the solvent from the tetrahydrofuran layer, and 1.5 g. the combined residues, yielded 0.7 g. (70%) VI, m. 183-5°. Heating 0.7 g. VI with 10 g. water in a 100 ml. flask, 4 hrs. at 80° in a pressure bottle, dilg. the mixt. with H₂O, extg. with Et₂O-C₆H₆, mixt., evap. the solvent, and h. at 100° the crude VII (10 g.) with 50 ml. 15% NaOH in MeOH in an autoclave 4 hrs. at 130-60° gave III after steam distn. and removal of the nontertiary amine, etc. (Ph. SO₂Cl); picrate, m. 241° (from MeOH), HCl, m. 274°; picrolonate, m. 224-5° (from EtOH). Neutralizing 1.5 g. HO₂CCH₂CMcEtCH₂CO₂H in 250 ml. H₂O with 25 ml. NH₄OH, heating the mixt. 4 hrs. at 80° in a 100 ml. flask, off the H₂O and NH₄, and crystg. the residue from Et₂O gave 113.5 g. (68%) 3-methyl-3-ethylglutarimide (XII), m. 120-6°. Extg. 120 g. XII in a Soxhlet app. with 100 ml. Et₂O, contg. 43 g. LiAlH₄, 16 hrs. yielded 4.5 g. (45%) VII (from the ether layer), and 37.5 g. (37.5%) VII.

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picrate, m. 163.5-5.5° (from H_2O). liquid deriv. in 71.2°
(from EtOH). Satg. a mixt. of 41 g. I. and petr. ether,
and 300 ml. 12% NaOH with Cl during 45 min. sepge the
petr. ether layer, washing it with 10% NaOH and
10% H_2SO_4 , drying with $CaCl_2$, treating with the steam of
85% H_2SO_4 while cooling with ice, sepge from the steam of
the chloramine in H_2SO_4 , illuminating the residue with
violet light 10 hrs., pouring the mixt. into water, treating
with NaOH, steam distg. the mixt. neutralizing the distillate
late 30.3 ml. 8N HCl, sepge the oil, washing with
residue after evap., with 30 ml. petr. ether, sepge the
NaOH wave, after steam distg. the residue, sepge the
picrate in 2% acetic MeOH, m. 125° (from EtOH).
EtOH, m. 163.5-5.5°.

21.9 g. II in 150 ml. petr. ether, sepge the oil, treating
and Cl under ultraviolet illumination, sepge the oil, treating
product with picric acid gave 11 g. of a solid, m. 200-3°
(dec. mpn) (from H_2O).
methadole, m. 333° (from acetone).
picrate (XIII), m. 363° (from petr. ether).
with HCl to give the chloride, treated with 20% H₂O₂ (for
degradation), and hydrogenation with 10% H₂SO₄ (for
methylethylpiperidine, picrate, m. 163.5-5.5°).
The result of the H₂O₂ degradation is a solid, m. 200-3°
IV for the product of the H₂O₂ degradation. M. 163.5-5.5°

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8. *picrate*, DI. 27A-73 *How many* *picrate* *is* *it*

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Distance by road is obtained by leaving mist of 0.5 m. ...
0 in. Mist in K. ...

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5(3)

PHASE I BOOK EXPLOITATION

CZECH/2479

Ferles, Miloslav, Engineer, Doctor, Polytechnic of Chemical Technology in Prague, Chair of Organic Chemistry; and Josef Jizba, Engineer, Doctor, Czechoslovak Academy of Sciences, Laboratory of Heterocyclic Compounds in Prague

Chemie pyridinu (Chemistry of Pyridine) Praha, Nakladatelství Československá Akademie Věd, 1957. 617 p. (Series: Československá Akademie Věd. Sekce chemická, sv. 21) Errata slip inserted. 1,300 copies printed.

Scientific Ed.: Rudolf Lukeš; Resp. Ed.: Věra Pošová; Tech. Ed.: Oldřich Dunka; Dedicated to Rudolf Lukeš, Academician.

PURPOSE: This book is intended for organic chemists specializing in aromatic heterocyclic compounds.

COVERAGE: This comprehensive work on pyridine is based on the literature of pyridine and its derivatives included in review periodicals from 1930 to 1953. The monograph was planned as a

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Chemistry (Cont.)

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supplement to Beilstein's handbook of organic chemistry, but in order to cover all possible pyridine derivatives, compounds with partly reduced pyridine nuclei and natural substances containing a pyridine ring or a partly saturated pyridine ring were included. Extensive bibliographies are given for each chapter and indicate wide coverage of Soviet and foreign literature including patent quotations. Maria Jizbová and Božena Ferlesová assisted in the preparation of the manuscript. Engineer Doctor Karl Bláh and Doctor of Medicine Vilma Bláhová wrote the chapters on the effects of pyridine. Engineer, Doctor Jiří Plíml reviewed the manuscript and Docent Engineer Doctor Miloš Hudlický was technical consultant. Candidates of Chemical Sciences Engineer Jiří Jary and Engineer Miroslav Pergál assisted in correcting the manuscript.

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FERLES, Miloslav

CZECHOSLOVAKIA/Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Referat Zhur-Khimiya, No 4, 1958, 11326.

Author : Ferles, M.

Inst :

Title : The Reduction of Quaternary Salts of Pyridine by Sodium Borohydride

Orig Pub: Chem Listy, 51, No 3, 474-478 (1957) (in Czech)

Abstract: The reduction of N-methylpyridinium halides (I); -2, -3, and -4-picolinium halides (II, III, and IV); and of -2,6-lutidinium halides (V) gives a mixture of tetrahydro- and hexahydro-derivatives whose composition is in agreement with the hypothesis that the reduction begins by the addition of a hydroxide anion at the 1,2-positions (formation of the tetrahydro derivative) or 1,4-positions (formation of the hexahydro derivative and cleavage of

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Tech Univ, Prague

CZECHOSLOVAKIA Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Referat Zhur-Khimiya, No 4, 1958, 11326.

the ring). The ratio of 1,2- to 1,4-addition is lower in 2-substituted pyridines and is increased in the case of 4-substituted pyridines. The substance and the ratio of 1,2- to 1,4-hydroxide ion addition are given below: I iodide, 4.3; I bromide, 4.1; II iodide, 2.8; II bromide, 1.9; III iodide, 4.1; IV iodide, 12.5; V iodide, 0.6. The above ratios were established on one hand by hydrogenation and on the other by the preparation of dibromide derivatives. The products are identified by comparison with pure samples. The results correspond to the reduction of HCOOH according to Lukesh et al (SZhKhim, 1957, 51256). Preparation: II iodide (for example) is dissolved in water, the solution is made alkaline with NaOH, and reduced by NaBH_4 during steam distillation. The total yield (82.8%) is determined by the titration of the distillate; the primary and secondary bases are removed

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CZECHOSLOVAKIA/Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Referat Zhur-Khimiya, No 4, 1958, 11326.

from the product by extraction with benzenesulfonyl chloride and the purified mixture of bases (a fraction) is hydrogenated over PtO_2 ; the picrate, mp 236° , is precipitated from the product. Another fraction is treated with Br_2 in HBr (acid) to form the 4,5-dibromodihydrobromide, mp 196° , from which the 1,2,5,6-tetrahydro base is precipitated with Zn ; picrate mp 227° . A similar synthesis procedure is used in preparing the picrates of the 1,2,5,6-tetrahydro derivatives of I (mp 201°), III (mp 105°), and IV (mp 146°). The picrates of the hexahydro derivatives of the following compounds have also been prepared: I, mp 221° ; III, mp $166-167^\circ$; IV, $181-182^\circ$. The following 3,4,-dibromo hydrobromides have been prepared: I, mp 191° ; III, mp 178° ; and IV, mp 176° . For comparison purposes 1-methylpiperidine was

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CZECHOSLOVAKIA/Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Referat Zhur-Khimiya, No 4, 1958, 11326.

synthesized by the reduction of N-methylglutarimide by LiAlH_4 by refluxing 18 hrs in ether, bp 103-105°/744 mm, yield 62%; picrate, mp 221°. 1,4-dimethylpiperidine was prepared by heating (200°, 2 hrs) 4-pipecoline hydrochloride with paraformaldehyde (picrate, mp 180°).

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FERLES, M

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CZECHOSLOVAKIA/Organic Chemistry. Synthetic Organic Chemistry.

Abs Jour: Ref. Zhur.-Khimiya, No II, 1958, 36216.

Author : Lukes R., Strouf O., Ferles, M.

Inst : Not given.

Title : On the Hofman's Decomposition of the Bicyclic Nuclei Containing Nitrogen in the Peripheral Positions. II. Breakdown of the Oxymethylate 7-Methyl-I-Azobicyclo (1,22)-Heptane.

Orig Pub: Chem. listy, 1957, 51, No 5, 923-926.

Abstract: Thermal decomposition of 7-methyl-I-azobicyclo-(1,2,2)-heptane (I) results in the exclusive formation of I-methyl-4-vinylpiperidine (II). The interaction between 4-acetyl-piperidine and CH_3Br in alcohol at approximately 20 forms methylate-bromide of 4-acetyl-piperidine (III) with 183-184° melting point (alc). The yield depends on the

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CZECHOSLOVAKIA/Organic Chemistry. Synthetic Organic Chemistry. G-2

Abs Jour: Ref: Zhur.-Khimiya, No II, 1958, 36216.

ing off a portion of C_6H_6 . The remaining solution is then boiled for 1 hour. The yield of methylatebromide I (Ia) thus obtained is 70.5%, melting point $304-305^\circ$; the melting point of picrate is $333-335^\circ$ (water). The water solution of Ia is mixed with the freshly prepared suspension of Ag_2O . A small quantity of $Ba(OH)_2$ water solution is added to the filtrate, followed by heating on a hot bath at 340° until dry. Water is added to the residue and the material is heated again until dry. Such an operation is repeated until the removed liquid is no longer alkaline. The distillate is then neutralized with dilute HCl , part of the solution is hydrated over Pt (as PtO_2), and I-methyl-4-ethylpiperidine (V) is removed in the form of picrate with melting point of $185.5-186^\circ$ (alc.), and also II, whose yield amounts to 50% and it has $146-146.5^\circ$

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CZECHOSLOVAKIA/Organic Chemistry. Synthetic Organic Chemistry.

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Abs Jour: Ref. Zhur-Khimiya, No II, 1958, 36216.

743mm boiling point, n_D^{20} of 1.4548, and d_4^{20} of 0.8365. The infra-red spectrum (1640 and 910 cm^{-1}) reveals presence of a vinyl group. Picrate II with $145.5 - 146^\circ$ melting point (from water) is identical with one described previously (Ref. Zhur.-Khimiya, 1956, 58059). For comparison, picrate V is obtained from chlorhydrine 4-ethylpiperidine and paraformaldehyde ($180-200^\circ$, 7 hours with the subsequent purification of the raw $n - \text{CH}_3\text{C}_6\text{H}_4\text{SO}_2\text{Cl}$), by hydration of 4-ethylpiperidine CH_3OH over a lattice type Ni ($140-185^\circ$, 6 hours, 145 atmospheres) with the subsequent purification of $n - \text{CH}_3\text{C}_6\text{H}_4\text{SO}_2\text{Cl}$. Refer to section I in the Ref. Zhur.-Khimiya, 1957, 47964.

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CZECHOSLOVAKIA/Organic Chemistry Synthetic Organic Chemistry. G-2

Abs Jour: Ref Zhur-Khim., No 24, 1958, 81674

Author : Lukes R , Strouf O , Ferles M.

Inst :

Title : The Hoffman's Cleavage of Bicyclic Bases Containing Nitrogen in the Side Radicals. II. The Splitting of the Oxymethylate of 7-methyl-1-azabicyclo (1,2,2) heptane.

Orig Pub: Collect. czechosl chem. commun , 1958, 23, No 2, 326-330.

Abstract: See R. Zh Khim , 1958, 36216.

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CZECHOSLOVAKIA / Organic Chemistry. Organic Synthesis. G-2

Abs Jour: Ref Zhur-Khimiya, No 10, 1959, 34888.

Author : Ferles, M.

Inst : Not given.

Title : Investigation of Pyridine Series. II. Reduction in Accordance with Ladenburg's Method and Electrolytic Reduction of Pyridine Bases.

Orig Pub: Chem. listy, 1958, 52, No 4, 668-673.

Abstract: Reduction of pyridines, picolins, and 2,6-lutidine either with sodium or electrolytically, in all the cases, results in a mixture of tetrahydrobases (I) and hexahydrobases (II). Since under the reduction conditions it is impossible to convert I into II, both products must be formed by different mechanisms. The reduction may start as a process of H₂ addition into 1, 2 or 1, 4

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CZECHOSLOVAKIA / Organic Chemistry. Organic Synthesis. G-2

Abs Jour: Ref Zhur-Khimiya, No 10, 1959, 34888.

Abstract: positions. The formation of Δ 1 and Δ 2-bonds leads to further reduction, Δ 3-bond does not reduce, therefore, the substitution in the 4 or 2 position results predominantly in the reduction of pyridins into II. In the former case I is formed, in the latter II. The reaction products are homogeneous even if isomerization of the position is possible: from 2-picoline is thus formed 2-methyl-1,2,3,6-tetrahydropyridine, from 3-picoline is formed 3-methyl-1,2,5,6-tetrahydropyridine. The reduction products of 2,6-lutidine have a cis-configuration. Reduction with sodium: a base in 20 times the quantity of C₄H₉OH is reduced with double the quantity of Na upon boiling for 2 hours followed by steam distillation. The distilled mixture of bases is treated (cold)

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CZECHOSLOVAKIA / Organic Chemistry. Organic Synthesis. G-2

Abs Jour: Ref Zhur-Khimiya, No 10, 1959, 34268.

Abstract: with bromine in HBr (acid) and from the obtained dibromide-- bromhydrate I is separated with the use of Zn. A sample is subjected to hydration in HBr over Pt followed by the determination of I quantity and from the product of hydration, II is separated. From 31 gr of 3-picoline are obtained 25.3 gr of distilled mixture that has 110-153°/749 mm boiling point and contains 28.4% I together with bromhydrate of 3,4-dibromo-3-methyl-piperidine (IV) of 170-171° melting point (from alc.), picrate of 3-methyl-1,2,5,6-tetrahydropyridine (V) of 161° melting point (from water), bromhydrate of 3-pipecoline (VI) of 162-163° melting point (from iso-C₃H₇OH). From 20 gr. of 4-picoline are also obtained 15.25 gr. of a 114-138°C/740 mm boiling point fraction that contains

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CZECHOSLOVAKIA / Organic Chemistry. Organic Synthesis. G-2

Abs Jour: Ref Zhur-Khimiya, No 10, 1959, 34888.

Abstract: 45% I, bromhydrate of 3,4-dibromo-4-methylpiperidino (VII) of 173-174° melting point (from CH₃OH), 4-methyl-1,2,3,6-tetrahydropyridine (VIII) of 135-136°/740 mm boiling point, and bromhydrate of 4-methylpiperidine (IX) of 173° melting point. Electrolytic reduction was conducted in 35% H₂SO₄ employing Pb electrodes and double-triple the quantity of current at 30 amp. level. Presented are: charge quantities, yields of mixed bases in %, contents of I in %, derived substances and their constants: pyridine, 58, 17, bromhydrate of 3,4-dibromopiperidino, melting point 200-201°, picrate of 1,2,3,6-tetrahydropyridine, melting point 160-162°; 2-picolino, 66, 24.4, bromhydrate of 4,5-dibromo-2-pipecolino, melting

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CZECHOSLOVAKIA / Organic Chemistry. Organic Synthesis. G-2

Abs Jour: Ref Zhur-Khimiya, No 10, 1959, 34888.

Abstract: point 202-203°, picrate of 2-methyl-1,2,3,6-tetrahydropiperidino (X base), melting point 160°; 3-picolino, 67, 22.4, IV, V, VI; 4-picolino, 58, 39, VII, VIII, IX; 2,6-lutidino, 76, 6, 20, bromhydrate of 3,4-dibromo-2,6-lupotidino, melting point 212°, ~~7~~¹⁰-2,6-lupotidino chlorhydrate, melting point 280-282°. 1,2,3,6-tetrahydropyridino under identical conditions (9 times the quantity of current) remains unchanged. X is heated for 5 hours up to 180-200° yielding 1,2-dimethyl-1,2,3,6 tetrahydropyridino picrate of 226° melting point. Analogically 1,4-dimethyl-1,2,3,6-tetrahydropyridine picrate of 146 melting point is obtained. The preceding Part can be found in Ref Zhur-Khimiya, 1958, 11326. -- Jan Kovar.

Card 5/5

G-30

CZECHOSLOVAKIA / Organic Chemistry. Organic Synthesis. G-2

Abs Jour: Ref Zhur-Khimiya, No 10, 1959, 34889.

Author : Ferlos, M.

Inst : Not given.

Title : Investigation of Pyridine Series. III. Reduction of the Quaternary Pyridine Salts Electrolytically and by Means of Lithium-Aluminum Hydride.

Orig Pub: Chem. listy, 1958, 52, No 4, 674-681.

Abstract: Results of the quaternary salts (QS) of pyridine bases with LiAlH_4 leads to the formation of corresponding N-alkyl substitutes of Δ^3 -tetrahydropyridines (I). In the electrolytic reduction of the same QS are formed mixtures of I with substituted piperidines (II). The COOR groups are reduced in the electrolysis up to CH_3 but under the action of LiAlH_4 , up to CH_2OH

Card 1/6

CZECHOSLOVAKIA / Organic Chemistry. Organic Synthesis. G-2

APPROVED FOR RELEASE: 08/23/2000

CIA-RDP86-00513R000412920001-2"

Abs Jour: Ref Zhur-Khimiya, No 10, 1959, 34889.

Abstract: groups. From QS of nicotinic acid wthyl ester (III) and $(\text{CH}_3)_2\text{SO}_4$ (III)-QS) the electrolytic reduction yields 1,3-dimethyl-1,2,3,6-tetrahydropyridine (V), which evidently is formed also in the reduction of 3-picolino iodomethylene with the aid of KBH_4 . 40 gr of pyridine and 63 gr. $(\text{CH}_3)_2\text{SO}_4$ are allowed to stand for 2 days followed by the addition of 35% H_2SO_4 up to the total volume of 250 ml. To 50 ml of the above solution are added 200 ml of 35% H_2SO_4 followed by the reduction with Pb cathode (200% of current). After the alkalization and steam distillation non-tertiary bases are separated with $\text{C}_6\text{H}_5\text{SO}_2\text{Cl}$ and 88.5% mixture of I and II (82% II) is derived; 1-methylpiperidine picrate of 221° melting point; 1-

Card 2/6

G-31

CZECHOSLOVAKIA / Organic Chemistry. Organic Synthesis. G-2

Abs Jour: Ref Zhur-Khimiya, No 10, 1959, 34889.

Abstract: methyl-3,4-dibromopiperidino bromhydrate of 191° melting point. Analogically are reduced QS of picolinos. From the QS of 2-picolino and (CH₃)₂SO₄ are obtained 76% II, 31.2% I, and derive 1,2-dimethyl-4,5-dibromopiperidino bromhydrate of 194° melting point and 1,2-dimethylpiperidino picrate of 236° melting point; from 3-picolino-iodomethylate 58.2% I, 1,3-dimethyl-3,4-dibromopiperidino-bromhydrate of 177.5 melting point, and 1,3-dimethylpiperidino-picrate of 166° melting point are obtained; from 4-picolino iodomethylate are derived 40% II, 18.9% I, 1,4-dimethyl-3,4-dibromopiperidino bromhydrate of 175° melting point, and 1,4-dimethyl piperidino picrate. After the electrolytic reduction of QS esters of piperidino carbonic acids mixtures are alkalized

Card 3/8

CZECHOSLOVAKIA / Organic Chemistry. Organic Synthesis. G-2

Abs Jour: Ref Zhur-Khimiya, No 10, 1959, 34889.

Abstract: with BaCO_3 and after the distillation of bases, Ba is precipitated with equivalent quantities of H_2SO_4 , followed by filtration, steaming, esterification by boiling with CH_3OH and HCl (gas), and separation of oils of the hydrated acids. III yields 25.7% of tertiary bases containing 89% I. 1,3-dimethylpiperidino-picrate, 1,3-dimethyl-4,5-dibromopiperidino bromhydrate of 167° melting point, 1,3-dimethyl-4,5-dibromopiperidino picrate of 177° melting point, IV, of 125/740 mm boiling point, picrate of 156° melting point, isodimethylate of 205° melting point are formed. From the methylsulfate of 1-methyl-4-carboethoxy-piperidinium (500% of current) are obtained 48.5% of tertiary bases, containing 48.6% I. Derived from the above are 1,4-dimethyl-1,2,3,6-tetrahy-

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G-32

CZECHOSLOVAKIA / Organic Chemistry. Organic Synthesis. G-2

Abs Jour: Ref Zhur-Khimiya, No 10, 1959, 34889.

Abstract: dropyridino-picrate of 146-147° melting point, picrate of 1,4-dimethyl piperidino of 181-182° melting point, and picrate of N-methylsonipocotinic acid of 160° melting point. From 1-methyl-4-carbomethoxypyridinium methylsulfate are derived (168% current) 31% of bases containing 75% I. Obtained are: methyl ester of 1-methylsonipocotinic acid of 90-93°/17 mm boiling point, 95-95.5°/22 mm, and picrate of 146-147° melting point. A solution of QS in CHCl₃ is added to LiAlH₄ in ether and the mixture is boiled. 3-methylpyridiniodomethylate yields 70% of tertiary bases containing 81% I. 5 gr. of iodomethylate of 4-methylpyridine in 20 ml CHCl₃ are reduced with 2 gr. LiAlH₄ for 3 hours, the yield of tertiary bases is 72%, containing 72% I; 3.8

Card 5/6

CZECHOSLOVAKIA / Organic Chemistry. Organic Synthesis. G-2

Abs Jour: Ref Zhur-Khimiya, No 10, 1959, 34889.

Abstract: gr of iodomethylate ester of iso-nicotinic acid (V ester) [obtained from 20 gr V in 40 ml C_6H_6 and 50 gr CH_3I (20° , 24 hours), yielding 33 gr of 121° melting point] is reduced with 1.2 gr $LiAlH_4$ for 6 hours, yielding 23% of 1-methyl-1-oxymethyl-1,2,3,6-tetrahydropyridine of $95-100^\circ/15$ mm boiling point; iodomethylate of 176.5° melting point. The reduction of iodomethylate with $NaBH_4$ in C_2H_5OH gave 50% yield of 1-methyl-4-carboethoxy-1,2,3,6-tetrahydropyridine of $108-109.5^\circ/15$ mm boiling point. -- Jan Kovar.

Card 6/6

C-33

AUTHOR: Ferles, M. CZECH/8-52-11-24/30

TITLE: Reduction of Some Nitrogen-containing Compounds with Sodium Aluminium Hydride (Redukce některých dusíkatých látek hydridem sodnohlinitým)

PERIODICAL: Chemické Listy, 1958, Vol 52, Nr 11, pp 2184 - 2185 (Czechoslovakia)

ABSTRACT: Selective reductions by sodium aluminium hydride were carried out on further nitrogenous derivatives. As an example of a Schiff's base benzalaniline (I) was reduced with sodium aluminium hydride; the awaited benzyl-aniline was produced. Reduction of cyclo hexanoneoxime (II) yielded cyclo-hexylamine. NaAlH_4 reduces cyclic imides and lactams to cyclic amines: 1-methyl-2-pyrrolidone III to 1-methyl-pyrrolidine; the methylimide of glutaric acid (IVa) gives 1 methyl-piperidine (Va) and the imide of β methyl- β -ethylglutaric acid (IVb) reduces to 4 methyl-4-ethylpiperidine (Vb). Sodium aluminium hydride behaves in the same way as lithium aluminium hydride; the advantage could be in its cheaper production costs.

Experimental: M.Pts are uncorrected, crystalline materials

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CZECH/8-52-11-24/30

Reduction of Some Nitrogen-containing Compounds with Sodium
Aluminium Hydride

were dried at 100 °C/1 mm for eight hours before analysis.
Reduction of benzalaniline (I). A suspension of NaAlH_4

(1.1 g) in dry tetrahydrofuran (150 ml.) was boiled for 3 hours, with mixing, with a solution of benzalaniline (3.8 g) in tetrahydrofuran (10 ml.). The reaction mixture, decomposed by dilute HCl , was steam distilled. The residual solution was made alkaline, shaken with ether; the ether solution dried (MgSO_4) - the ether evaporated.

The only residue solidified in a crystalline mass (1.8 g)
M.Pts - found: 36, literature: 37-38° for benzyalaniline.

Reduction of cyclohexanone oxime (II)

A cyclohexanone oxime solution in tetra hydrofuran (3 g in 50 ml.) was added to a suspension of NaAlH_4 in tetrahydrofuran (3 g in 150 ml.) and the reaction mixture boiled, with stirring, for 3 hours. The yellow-coloured contents of the flask were decomposed by 10% H_2SO_4 , the solution steam distilled, then made alkaline

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CZECH/8-52-11-24/30

Reduction of Some Nitrogen-containing Compounds with Sodium Aluminium Hydride

and steam distilled once more. The distillate was neutralised by 1.04 N.HCl (16.4 ml. = 62% base). The solution of the hydrochloride was mixed with benzoylchloride (3.5 g); a sufficient quantity of caustic soda and the reaction mixture was shaken. The crystals produced were filtered by suction M.Pt. 145° (benzene). Literature gives M.Pt 147° for benzoylamino cyclohexane.
For $C_{13}H_{17}ON(203.3)$ - Calculated: 76.81% C, 8.43% H, 6.89% N
Found: 77.06% C, 8.26% H, 6.78% N

Reduction of 1-methyl-2 pyrrolidone (III)

A mixture of $NaAlH_4$ (1 g), tetrahydrofuran (100 ml.) and a solution of 1-methyl-2 pyrrolidone in tetrahydrofuran (2 g in 20 ml.) were boiled, with stirring, for 8 hours. The flask contents were worked up as in the preceding section, with the exception that after neutralisation of the distillate (18.2 ml., 1.04 N-HCl, 94% base) the solution of the hydrochloride was mixed with benzene sulphochloride (5 g) and caustic soda and the mixture shaken for two hours. The reaction mixture was steam

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Reduction of Some Nitrogen-containing Compounds with Sodium Aluminium Hydride

distilled, the distillate neutralised (16.9 ml., 1.04 N-HCl, 87% tertiary base). The picrate was prepared from the hydrochloric solution M.pt. 218-219° (water) as in the literature. The substance gives no depression of M.Pt with authentic 1-methyl-pyrrolidine picrate.
 $C_{11}H_{14}O_7N_4$ (314.3) Calculated: 42.04% C, 4.49% H, 17.83% N
 Found: 41.80% C, 4.51% H, 18.01% N.

Reduction of the methylimide of glutaric acid (IVa)

A mixture of $NaAlH_4$ (2 g) tetrahydrofuran (150 ml.) and methylglutarimide (2.5 g) was boiled, with stirring, for seven hours. After working up the reaction mixture as in the preceding section: yield of picrate 4 g (62%) M.Pt 220° C (water) which agrees with the information given for 1-methyl-piperidine picrate:
 $C_{12}H_{16}O_7N_4$ (328.3) Calculated: 43.90% C, 4.91% H, 17.07% N
 Found: 43.77% C, 5.19% H, 16.90% N

Reduction of the Imide of β -methyl-ethylglutaric Acid (IVb)
 The imide of β -methyl- β ethyl glutaric acid (3.1 g) was reduced by boiling with $NaAlH_4$ in tetrahydrofuran

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(2.2 g in 200 ml.). The hydrochloride was prepared from the reaction mixture distillate after making it alkaline (6 ml., 1.18 N-HCl, 28% base) and from this hydrochloride the picrate was prepared M.Pt 165 °C (water) which melted, without depression, with 4-methyl-4-ethylpiperidine picrate. $C_{14}H_{20}O_7N_4$ (356.3) Calculated: 47.18% C, 5.66% H, 15.72% N
Found: 47.05% C, 5.88% H, 15.51% N.

There are 8 references, 4 of which are Czech, 1 German and 3 English.

ASSOCIATION: Katedra organické chemie, Vysoká škola chemicko-technologická, Praha (Department of Organic Chemistry, Chemico-technological Technical University, Prague)

SUBMITTED: February 21, 1958

This is a complete translation, except for acknowledgments, structural formulae and references.

Card 5/5

FERLES, M.; STROUF, O.; LUKES, R.

"Hofmann degradation of bicyclic bases with nitrogen on the border of rings"

III. Degradation of methoxyhydroxide of 7-methyl-1-aza-bicyclo-(1,2,2)-heptane.
In German. p. 212.

COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS, Praha, Czech.,
Vol. 24, No. 1, Jan. 1959

Monthly List of East European Accessions (EEAI), LC, Vol. 8, No. 6, Sept. 59
Unclassified

FERLES, M

Pyridine series. IV. Electrolytic reduction of pyridine carboxylic acids. M. Ferles and M. Krstina (Vysoká škola chem. technol., Prague) Collection Czechoslov. Chem. Commun. 34, 2325-34 (1969) (in German); cf. C.A. 53, 20054c; Sorn, C.A. 42, 7298a. — Electrolytic reduction of picolinic acid (I) [prepd. from 61.3 g. Et ester (II)] in 89% H_2SO_4 at 20° on activated Pb electrodes (230 amp. hrs., current density 7.4 amp. per dm²) yielded 2-picoline, 2-current density 7.4 amp. per dm²) yielded 2-picoline, 2-tetrahydropyridine (III) (picrate m. 155° (AcORt)), and very small amounts. (detection by paper chromatography) of piperidine (IV); methylation of the III HCl salt with (HCHO), at 185° for 3 hrs. gave 1,2-dimethyl-1,2,3,6-tetrahydropyridine (V); picrate m. 227° (H₂O). Redn. (200 amp. hrs.) of II gave similar results. Redn. (216 amp. hrs.) of II methanesulfate [prepd. from 51 g. II and 42.6 g. Me_2SO ; an oil] in 250 ml. 30% H_2SO_4 yielded an unsatd. base (probably V), 1,2-dimethylpiperidine (picrate m. 215° (H₂O)), 1-methylpiperidine acid (N-methylpiperidine, VIII). Redn. (400 amp. hrs.) of 181 g. Et ester (II) in 250 ml. 80% H_2SO_4 yielded 7.5 g. guanine HCl salt, m. 300° (H₂O), and 13.2 g. mixt. of bases, bps 124-6.5°, identified as 2-methyl-1,2,3,6-tetrahydropyridine (IX) (picrate m. 180° (AcORt)), 2-methyl-1,2,3,6-tetrahydropyridine (X) (picrate m. 180° (AcORt)), and 2-methylpiperidine;

picrate m. 137° (EtOH); HBr salt m. 161° (EtOH). Picrates of IX and X formed a eutectic, m. 145°. Adding dropwise 12.5 g. II methiodide (XI) in 40 ml. anhyd. $CHCl_3$ to 4 g. $LiAlH_4$ in 300 ml. Et₂O, refluxing the mixt. with agitation 6 hrs., and decamp. with 4 ml. H_2O , 4 ml. 15% NaOH, and 12 ml. H_2O gave 2.65 g. 1-methyl-2-hydroxymethyl-1,2,3,6-tetrahydropyridine (XII), a very unstable oil, b. 78°; methiodide m. 255° (EtOH). Hydrogenation of XII in EtOH over PtO_2 gave 1-methyl-2-hydroxymethylpiperidine (XIII), b. 85°; picrate m. 185-9° (EtOH). Refluxing 1 hr. 2 g. 2-hydroxymethylpiperidine (XIV), 10 ml. MeOH, 15 ml. C_6H_6 , and 10 g. MeI gave 2.8 g. XIV, methiodide (XV), m. 183° (MeOH- C_6H_6). Hydrogenating 12 hrs. 1.5 g. XV and 10 ml. 50% aq. MeOH (0.1 g. PtO_2) gave XIII, isolated as the picrate. Adding in 20 min. with agitation 3.4 g. NaBH₄ to 36 g. XI in 300 ml. abs. EtOH, stirring the mixt. 1 hr., evap. in vacuo, dissolving the residue in H_2O , adding Na_2CO_3 , extg. with Et₂O, drying the exts. with K_2CO_3 , and distg. gave 11 g. very unstable VIII Et ester (XVI), b. 83-92°. Hydrogenation of XVI in EtOH over PtO_2 followed by hydrolysis with 20% aq. HCl gave VII. Hydrolysis of XVI with H_2O gave syrupy VIII. Redn. of 1.27 g. as the HCl salt, m. 160° (EtOH-Et₂O). Redn. in 50 ml. Et₂O of XVI in 20 ml. Et₂O with 0.6 g. $LiAlH_4$ in 50 ml. Et₂O gave 0.42 g. XII. Adding dropwise at 0° 2.37 g. XII to 10 ml. $SOCl_2$, refluxing the mixt. 1 hr., evap. the excess $SOCl_2$ in vacuo, dissolving the residue (3.5 g.) in 25 ml. $CHCl_3$, adding dropwise 2.5 g. $LiAlH_4$ in 100 ml. Et₂O, and refluxing

M. Ferles and M. Prystas

the mixt. 6 hrs. on a steam bath gave 77% V, isolated as the picrate, m. 226° (H₂O). Adding at 0° 11 g. XVI in 60 ml. Et₂O to 7 g. BrCN in 50 ml. Et₂O, keeping the mixt. 24 hrs. at 20°, refluxing 1 hr. sepg. the Et₂O layer, washing with N HCl, H₂O, and aq. NaHCO₃, drying with K₂CO₃, and distg. gave 1.7 g. Et 1-cyano-1,2,3,6-tetrahydropicolinate (XVIII), b_p 120°, a very unstable oil. Hydrolysis of 1 g. XVIII with 20 ml. 2.5N HCl for 3 hrs. (reflux) gave 35% IV Et ester, b_p 95°. An analogous demethylation of 1.8 g. 1,3-dimethyl-1,2,3,6-tetrahydropyridine in 35 ml. Et₂O with 1.2 g. BrCN in 40 ml. Et₂O gave 0.3 g. 1-cyano-3-methyl-1,2,3,6-tetrahydropyridine, b_p 112°, whose hydrolysis yielded IX, isolated as the picrate, m. 187°. Pure amino acids prepd. by electrolysis, as well as synthetically were chromatographed (ascending technique) on Whatman No. 1 paper in 4:1:1 BuOH-AcOH-H₂O and developed with ninhydrin (secondary amino acids) or iodine vapors (tertiary amino acids); R_f values given: I, 0.87; pipecolic acid, 0.82; IV, 0.89; VI, 0.85; VIII, 0.87.

JHM-20002

FEB 22, 1961

V Reduction of pyridine bases with formic acid. XI, 3-(2-pyridyl)acrylic and 3-(4-pyridyl)acrylic acids. R. Lukat, J. N. Zvonkova, A. P. Lukat, *Zh. Obshch. Khim. Commun.* 25, 2688-74 (1951).—Redn of 3-(2-pyridyl)acrylic acid (I) with HCO_2H gave 3-(2-pyridyl)propionic acid (V) and 2-(2-pyridyl)propionic acid (VI). 3-(2-pyridyl)acrylic acid (I) with HCO_2H in 2.5 l. xylene 12 hrs. at 150° , then 30% HCl, and alkalinizing the ext. with NaOH gave 3-(2-pyridyl)propionic acid (V) and 2-(2-pyridyl)propionic acid (VI). This acid, on treatment with MeI in Me_2CO , to Et 3-(2-pyridyl)acrylate methiodide (VIII), m. $157-9^\circ$ (Me_2CO), 45% yield. Treatment of 89 g. VIII in 300 ml. H_2O with Ag 2O prepd. from 80 g. AgNO_3 afforded 40 g. (80%) I, m. $162-3^\circ$ ($\text{MeOH}-\text{Me}_2\text{CO}$). Refluxing 40 g. I with 120 ml. 95% HCO_2H and 200 g. fused HCO_2K 13 hrs. at $143-8^\circ$, alkalinizing the mixt. with KOH, steam, distg. the MeNH_2 formed, acidifying the residue with HCl, filtering with activated charcoal, evapg. the filtrate *in vacuo*, treating the residue with 200 ml. abs. EtOH, satg. 5 hrs. with HCl, dissolving the residue in 200 ml. CHCl_3 , adding NH_3 in CHCl_3 , removing the NH_4Cl , and distg. gave 4.2 g. (9.5%) Et ester (IIa) of 3-(2-pyridyl)acrylic acid, b. $94-5^\circ/10$ l. EtOH- H_2O ; methiodide (XI), m. $94-5^\circ$. Et ester of III, b. $105-7^\circ$; methiodide (XII), m. $105-7^\circ$. Et ester (IIa) also obtained by hydrogenation of I over PtO_2 , esterification, and treatment with formic acid, m. $114-16^\circ$. Refluxing a stirred mixt. of 24 g. 3-(2-pyridyl)acrylic acid, 9 g. P, 120 ml. AcOH, and 160 ml. MeOH 14 hrs., dilg. the mixt. with 160 ml. H_2O ,

filtering, evapg. the filtrate *in vacuo*, washing the cryst. residue (24 g., m. $155-7^\circ$) with Me_2CO , dissolving it in 50 ml. hot H_2O , treating the soln. with a sat. 10% NaH_2PO_4 , evapg. the mixt. *in vacuo*, boiling the residue with 20 ml. BuOH, filtering, and cooling the filtrate gave 13 g. (53.8%) II in $143-4^\circ$. Reducing 13 g. II with 80 ml. EtOH and 5 ml. H_2SO_4 4 hrs. evapg. the EtOH *in vacuo*, dissolving the residue in 50 ml. CHCl_3 , adding NH_3 in CHCl_3 , removing the NH_4Cl , and distg. gave 1.8 g. (13.8%) Et ester (IIa). Et ester (IIa) was also obtained in 65% yield by hydrogenation of I over PtO_2 in AcOH, reducing 4.2 g. (9.5%) Et ester (IIa) over PtO_2 in AcOH, prepd. from 1,1,1-trichloro-3-(4-pyridyl)propanol in 36% yield; with 120 ml. EtOH and 20 ml. H_2SO_4 4 hrs. on the steam bath, evapg. the EtOH, alkalinizing the residue with Na_2CO_3 , and extg. with EtOH gave 151 g. (85%) Et 4-(4-pyridyl)acrylate, m. 94° , methiodide (IX), m. $170-1^\circ$ (EtOH). Treatment of 279 g. IX in 2 l. H_2O with Ag_2O (from 175 g. AgNO_3) afforded 168 g. (97%) IV, m. $85-6^\circ$. Refluxing 30 g. IV with 150 ml. 95% HCO_2H and 167 g. fused HCO_2K 10 hrs. at $156-7^\circ$, alkalinizing the mixt., steam-distg. the MeNH_2 formed, acidifying the residue with HCl, filtering off the KCl, evapg. the filtrate *in vacuo*, dissolving the residue (after drying) in 500 ml. MeOH, satg. the mixt. during 3 hrs. with HCl, keeping 4 hrs. on the steam bath, refluxing, distg. the solvent, dissolving the residue in CHCl_3 , treating the soln. with NH_3 in CHCl_3 , filtering off the salt, and distg. the filtrate gave 0.9 g. Me ester (X) of V, b. 113° , picrate m. $110-2^\circ$ MeOH; picrate of Me ester of VI, m. $170-1^\circ$ MeOH. X, b. 113° , was also prepd. in 80% yield by treatment of V with CH_3I , methiodide (XI), m. $98-9^\circ$ Me 2CO . The Me ester of VI was also obtained by treatment of 1.4 g. XI in 40 ml. MeOH with 5 g. NaBH_4 , b. $89-90^\circ$, 50% yield. Hydrogenation over PtO_2 in MeOH afforded Me 3-(1-methyl-4-piperidyl)propionate, b. $80-2^\circ$; picrate m. $197.5-8.5^\circ$ (MeOH). M. Hudlický

Z/008/61/000/001/001/005
E112/E253

AUTHORS: Bláha, K. and Ferles, M.
TITLE: Academician Rudolf Lukeš. (Obituary)
PERIODICAL: Chemické listy, 1961, No. 1, pp. 1-15 + 2 plates
TEXT: Academician Rudolf Lukeš, D.Sc. who died on October 17, 1960 was Professor of Organic Chemistry at the University of Chemical Technology, Prague, President of the Czechoslovak Chemical Society at the Czechoslovak Academy of Sciences and Director of the Laboratory for Heterocyclic Compounds at the same institution. The present paper is an appraisal of his scientific and pedagogic achievements. Starting his career as a theoretical sugar chemist with E. Votoček, he concentrated later on the chemistry of heterocyclic compounds, this work being initiated by his discovery of the reaction of N-succinimide and N-methylglutarimide with the Grignard reagent. This method permits the lengthening of aliphatic acid chains by 4 or 5 carbons and makes readily available a number of new heterocycles, e.g. pyrrolones. It was shown by Lukeš that the two carbonyl groups in the cyclic imides display different functionality, and that only

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one of them was capable of reacting with the Grignard reagent. During the German occupation of the country and the closure of the Czech universities, Lukeš worked for the Society for Chemical and Metallurgical Production, Prague, where he was concerned with the chemistry of furane and discovered an electrolytic method for the reduction of keto-di-carboxylic acids (reduction of chelidonic- to pimelic acid). A new reaction was also discovered during the electrolytic reduction of dilevulinic acid: In addition to the expected sebacic acid, valeric acid also formed by fission of -C-C-chain between both carbonyl groups. In the field of furane chemistry, the 2,5-dicarboxylic acid was reacted with ethylene glycol to produce fibre forming monomers, similar to the poly-ethylene terephthalates. Work in the field of heterocycles, carried out at the above firm produced interesting results by the reduction of quaternary pyridinium compounds with formic acids, leading to piperidine and Δ^3 -piperidines. The latter products, which, so far, were not easily accessible, opened the way for an easy synthesis of a number of pyrrole derivatives. New methods

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Academician Rudolf Lukeš. (Obituary)

were evolved to isolate and characterize the new bases and Lukeš found that β -alkyl-substituted derivatives could be isolated as quaternary salts with dinitro-chlorobenzene. The last period of Lukeš' activities were occupied with the study of alkaloids and particularly with problems of their stereochemical relationship and biological syntheses. Preparatory work consisted in establishing the relative configuration of some aminoalcohol, followed by determining the absolute configuration of a number of alkaloids (sedamine, hygrine, anabasine, etc.) The stereochemical course of many reactions, which could be taken as model substances for the understanding of biological alkaloid syntheses, was investigated. The work has not been completed. Lukeš and collaborators were the authors of text-books on Organic Chemistry (2 volumes) and Fundamentals of Preparative Organic Chemistry (1 volume). A list of original papers (189), patents (2), preliminary communications (35), lectures in print (7), books and manuscripts (12) and others (7) is appended.

Card 3/3

CZECHOSLOVAKIA

FERIES, M; CAPLOVIC, J.

Institute of Organic Chemistry of the Technical High School
of Chemistry, Prague (for both)

Prague, Collection of Czechoslovak Chemical Communications,
No 6, 1963, pp 1434-1439

"Studies in the Pyridin Series VIII. On the High Pressure
Hydration of Nicotinic Acidic and Isonicotinic Acidic
Ethylester."

FERLES, M.

"Preparative reactions in organic chemistry" by J. Novak,
J. Zemlicka. Pt. 7: "Catalyzed acid syntheses," Reviewed by
M. Ferles. Chem listy 57 no. 3: 279-280 Mr '63.

PERLES, M.

Studies in the pyridine series. Pt. 9. Coll Cz Chem 29 no.10:
2323-2327 O '64.

1. Department of Organic Chemistry, Institute of Chemical
Technology, Prague.

FERLES, M.

"Chemistry of heterocyclic compounds" by A.Weissberger.
Reviewed by M.Ferles. Chem listy 58 no.11:1357-1358 N
'64.

FERLES, Miloslav

"Publications in organic syntheses" by Jee- Mathieu, Andre
Allais. Reviewed by Miloslav Ferles. Chem prum 15 no.1:
62 Ja '65.

1. Chair of Organic Chemistry of the Higher School of
Chemical Technology, Prague.

CZECHOSLOVAKIA

FERLES, M.

JANAK, J.; HOLIS, E.; FERLES, E.

1. Institute for Inorganic Chemistry, Technical Higher School for Chemistry (Institut für organische Chemie, Technische Hochschule für Chemie), Prague (for Janak);
2. Institute for Instrumental Chemical Analysis, Czechoslovak Academy of Sciences (Institut für instrumentale analytische Chemie, Tschechoslowakische Akademie der Wissenschaften), Brno (for ?)

Prague, Collection of Czechoslovak Chemical Communications, No. 3, March 1966, pp 1273-1280

"Investigation of the piperidine series. Part 10: Gas chromatographic separation of some position isomerisms of the piperidine series."

CZECHOSLOVAKIA

PERLES, M; KOVARIK, M; VONDRACKOVA, Z

Institute of Organic Chemistry, Technical College of
Chemistry (Institut fur organische Chemie, Technische
Hochschule fur Chemie), Prague - (for all)

Prague, Collection of Czechoslovak Chemical Communi-
cations, No 3, March 1966, pp 1348-1354

"Investigation of the pyridine series. Part II: Reduction
of quaternary salts of some pyridine homologues with
sodium borohydride."

OZECHOSLOVAKIA

FERLES, M; JANKOVSKY, M

1. Department of Organic Chemistry, Institute of Chemical Technology, Prague - (for ?). 2: Department of Chemistry, Institute of Agriculture, Prague-Suchdol - (for ?)

Prague, Collection of Czechoslovak Chemical Communications,
No 7, July 1966, pp 3008-3001

"Studies in the pyridine series. Part 14: Novel procedures
of preparing 3-butylisonicotinic acid."

(3)

CZECHOSLOVAKIA

KSANDR, Z; SAMEK, Z; SPIRKO, V; FERLES, M

1. Department of Analytical Chemistry - (for ?). 2: Department of Organic Chemistry - (for ?), Institute of Chemical Technology. 3: Institute of Organic Chemistry and Biochemistry, - (for ?). Czechoslovak Academy of Sciences, Prague

Prague, Collection of Czechoslovak Chemical Communications,
No 7, July 1966, pp 3003-3007

"Studies in the pyridine series. Part 13: NMR-shift ranges for some isomeric alkylsubstituted tetrahydropyridines."

CZECHOSLOVAKIA

FERLES, M; HOLIK, M

Institute of Organic Chemistry, College of Chemical
Engineering, Prague - (for both)

Prague, Collection of Czechoslovak Chemical Communi-
cations, No 1, January 1967, pp 457-460

"Investigation of the pyridine series. Part 16:
Wittig's reaction with some 1-methyl piperidones."

PERIUGA, Cvjeteta, dr.; HELLENBACH, Helena, dr.

Epidemic of serous meningitis in Zagreb in 1956. Liječ. vjes. 81
no. 11: 811-818 '59.

1. Iz Bolnice za zarazne bolesti u Zagrebu.
(MENINGITIS epidemiol.)

MIHALJEVIC, Frane, dr.; BACUN, Marija, dr.; ~~HEJZAR~~, Branko, dr.; FERLUGA,
Cvijeta, dr.; HELLENBACH, Helena, dr; KNEZEVIC, Mira, dr.;
KOSUTIC, Zvonko, dr.

Hornholm disease in Yugoslavia. Liječn. vjesn. 83 no.8:771-781
'61.

1. Iz Bolnice za zarazne bolesti u Zagrebu.
(PLEURODYNIA EPIDEMIC epidemiol)

I 39006-66 T JK

ACC NR: AP6029579

SOURCE CODE: YU/0015/65/000/08-/0185/0188

AUTHOR: Mihaljevic, Fran (Professor; Doctor); Ferluga, Cvijeta (Doctor);
Schonwald, Slavko (Doctor) 17B

ORG: Infectious Diseases Hospital/directed by Professor, Doctor F. Mihaljevic/,
Zagreb (Bolnica za zarazne bolesti)

TITLE: Hemorrhagic fever with renal syndrome

SOURCE: Medicinski glasnik, no. 8-9, 1965, 185-188

TOPIC TAGS: diagnostic medicine, gland, biologic metabolism, physiologic parameter

ABSTRACT: Data on 5 patients treated since 1952, and selected because considered the "most typical" among many: case histories, graphs showing course in individual patients; tabulation of 18 symptoms or signs or findings in the 5. Differential diagnosis discussion. [Based on authors' Eng. abst.] [JPRS: 36,599]

SUB CODE: 06 / SUBM DATE: none / SOV REF: 002 / OTH REF: 003

Card 1/1 HS

0917 2673

KOSUTA, S.; KRZIC, M.; FERLUGA, D.

Fat embolism in liver steatosis. Zdrav. vestn. 34 no.5/6:100-104
'65.

1. Interna klinika medicinske fakultete v Ljubljani (predstojnik:
prof. dr. S. Mahkota) i Patolosko-anatomski institut medicinske
fakultete v Ljubljani (predstojnik: prof. dr. F. Hribar).

FERLUGA, D.; PREZELJ, F.

Atresia of the tricuspid ostium. Zdrav. vestn. 34 no.5/6:
105-110 '65.

1. Interna klinika medicinske fakultete v Ljubljani (pred-
stojnik: prof. dr. S. Mahkota) i Patolosko-anatomski institut
medicinske fakultete v Ljubljani (predstojnik: prof. dr.
P. Hribar).

FERLJUGA, Dusan

Statistical study on 304 autopsies in diabetes. Zdrav. vestn. 33
no.3:81-84 '64

1. Patolosko-anatomski institut medicinske fakultete v Ljubljani
(Predstojnik: prof. dr. France Hribar).

NIKULIN, A.; FERLUGA, J.; STERN, P.

The influence of the tissular histamine level on inflammatory processes.
Acta med. iugosl. 16 no.2:182-188 '62.

1. Institut za patolosku anatomiju i Institut za farmakologiju
Medicinskog fakulteta u Sarajevu.
(HISTAMINE) (INFLAMMATION)

FERLUGA, J.

On the anaphylatoxin. Bul se Young 7 no.1/2:11 F-Ap '62.

1. Zavod za farmakologiju Medicinskog fakulteta, Sarajevo.

*

FERMANOZHIEV, I.

"Electric cone test as a tool for finding oil structures" (p.49) PRIRODA
(Bulgaraska Akademia Na Naukite) Sofiya Vol 3 No 1 Jan/Feb 1954

SO: East European Accessions List Vol 2 No 6 Aug 1954

FERMANDZHIEV, I.

"Relief influences on the earth's magnetic field."

IZVESTIIA. SERIIA FIZICHESKA, Sofia, Bulgaria, Vol. 6, Jan./Dec. 1956
(published 1957).

Monthly List of East European Accessions Index (EEAI), The Library of
Congress, Volume 8, No. 8, August 1959.

Unclassified

FERMANDZHIEV, I.

Electric field of vertical polarized column in anisotropic surrounding, p. 199.

GODISHNIK. MATEMATIKA I FIZIKA. Sofia, Bulgaria, Vol. 50, no. 1 pt. 2

Monthly List of East Accession (EEAI) LC, Vol. 9, No. 1 January 1960

Uncl.

CZECHOSLOVAKIA/Human and Animal Morphology - The Skeleton:

S

Abs Jour : Ref Zhur Biol., No 5, 1959, 21555

Author : Formanek, G., Fernar, H.

Inst : -

Title : Bone Changes in Hemophilia

Orig Pub : Ceskosl. rentgenol., 1957, 11, No 2, 120-126

Abstract : No abstract.

Card 1/1

FERMI, Enrico.

On the origin of cosmic radiation. Magyar fizikai folyoir 8 no.2:149-160
'60. (EEAI 9:10)

1. A Chicagói Egyetem Magfizikai Intézete, Chicago, Illinois.
(Cosmic rays)

[illegible]

FERMOR, N. A.

*General and
Physical Chemistry*

Concerning S. S. Voronko's and M. A. Zaitseva's article
"Mutual solubility and its significance in emulsion poly-
merization." N. I. Smirnov and N. A. Fermor. *Uspekhi*
Khim. 41, 368 (1963). — Criticism of V. and Z. (*C.A.* 41,
5006d) in respect to arithmetical errors which give erroneous
impressions of variation of reaction rates with temp. changes.
G. M. Kosolapoff

MF 7-27-54

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FERMOR, N.A.; PEYZNER, A.B.

Mechanism of emulsion polymerization. Part 1. Effect of the nature of the cation of anion-active emulsifiers, on polymerization of styrene. Koll. zhur. 15 no.4:292-298 '53. (MLRA 6:8)

1. Nauchno-issledovatel'skiy institut sinteticheskogo kauchuka imeni S.V. Lebedeva (Leningrad). (Polymers and polymerization)
(Styrene)

The mechanism of emission of light

the polymerization of styrene N. A. Serebrennikov

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2. This mechanism of emulsion polymerization. II. Calculation of the composition of the particles in potassium oleate and ammonium oleate solutions. N. A. Ferman and A. B. Pelzner (S. V. Lebedev Sci. Research Inst. Synthetic Rubber, Leningrad). *Kolloid. Zhur.* 15, 459-55 (1953); cf. *C.A.* 47, 11794g. From the elec. cond. and the f.p. depression the compn. of aq. solns. of K oleate (I) and NH₄ oleate (II) was calcd. by McBain's method. E.g., a 0.145N I soln. at pH 7 contained 0.0034, 0.0043, 0.0037, 0.0243, and 0.1097 g. equiv. of simple ions, simple mol., ionic micelles, neutral colloid, and acid colloid, resp. At pH 12.1 these quantities ($\times 10^4$) were 10, 41, 202, 1213, and 0, resp. In 0.145N II soln. at pH 7 the figures were 15, 10, 11, 318, and 1100×10^{-4} , and at pH 10.2, 3, 5, 124, 1300, and 45×10^{-4} . The elec. cond. at 18° at the ratio K:oleic acid = 1:1 was 0.0046, and at NH₄:oleic acid = 1:1 it was 0.0083.

J. J. Bikerman

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USSR .

The mechanism of emulsion polymerization. III. The effect of the charge of the colloidal part of soap solution on the rate of polymerization of styrene in emulsion. V. A. Petrov and A. B. Pecher. *Colloid J. (USSR)* 16, 231-236 (1954) (Engl. translation). See C.A. 48, 13552. H. L. H.

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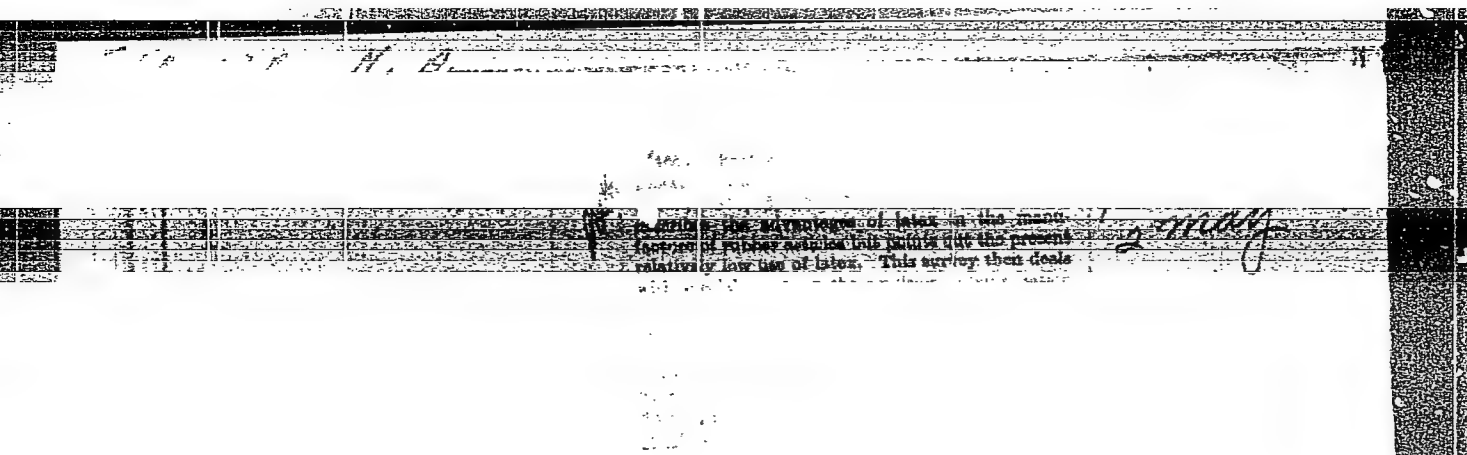
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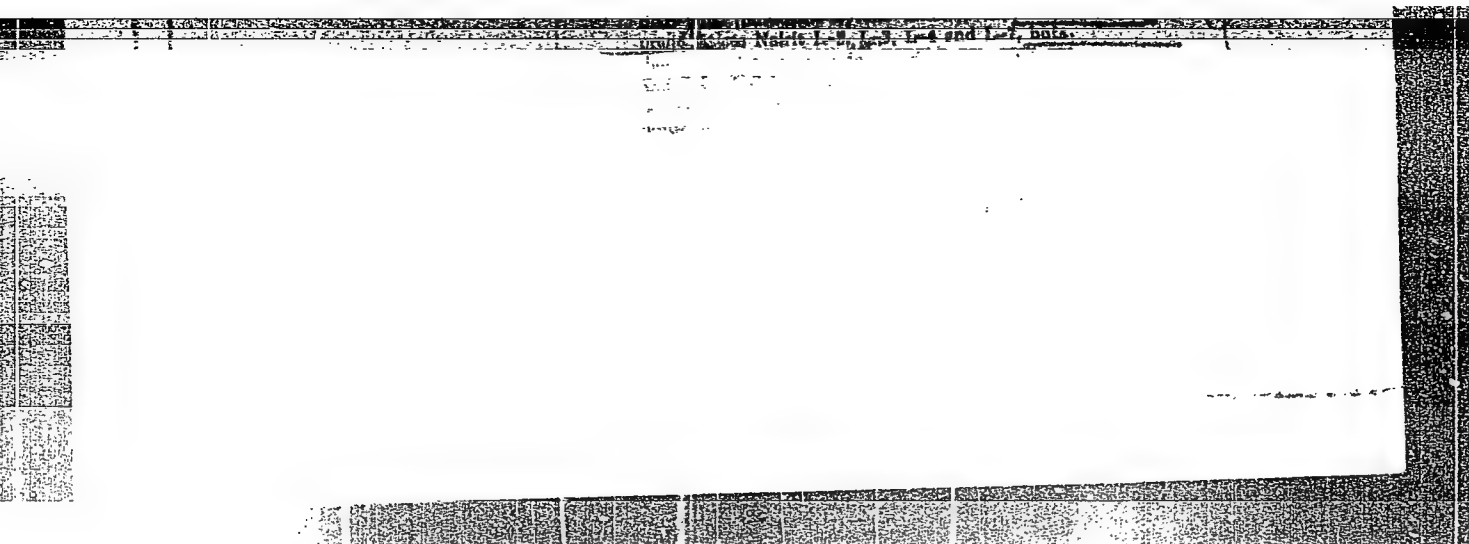
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FERMOR, M.A.; PEYZNER, A.B.

Synthetic latex used for cable insulation. Biul.tekh.-ekon.inform.
no.11:11-13 '58. (MIRA 11:12)
(Latex) . (Electric insulators and insulation)

SOV/138 -58-4-2/13

AUTHORS: Peyzner, A. B; Fermor, N. A; Lebedev, A. V.

TITLE: On the Influence of Plasticity of Rubber on the Technological Properties of Synthetic Latexes. (O vliyani plastichnosti kauchuka na tekhnologicheskiye svoystva sinteticheskikh lateksov)

PERIODICAL: Kauchuk i Rezina, 1958, ¹⁷Nr.4. pp. 4 - 5. (USSR).

ABSTRACT: S. V. Lebedev (Ref.1) showed that the plasticity is an important property of sodium 1,3-butadiene rubber, and, therefore, the plasticity was taken as a basis for classifying different types of this rubber (Ref.2). The plasticity indicates the basic molecular characteristics of the rubber (average molecular weight, fractional composition, branching etc.). It is, therefore, necessary to regulate the plasticity of emulsion rubbers during their synthesis. There are some publications on the synthesis of latexes containing rubbers of various plasticity (Ref.3); but no systematic investigations have been carried out on their use for the manufacture of various goods. The plasticity of the rubbers was proved to be one of the essential factors during investigations carried out by the All-Union Research Institute for Synthetic Rubber (VNIISK) (Vsesoyuznyy

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SOV/138--58-4-2/13

On the Influence of Plasticity of Rubber on the Technological Properties of Synthetic Latexes.

nauchno-issledovatel'skiy institut sinteticheskogo kauchuka (VNIISK) on the conditions for the manufacture of industrial latexes. VNIISK, NIIR (Research Institute for Rubber and Latex Goods) (Nauchno-issledovatel'skiy institut rezinovykh i lateksnykh izdeliy) and the Leningrad Factory RTI elaborated methods and conditions for the preparation of special latexes SKS-50 PG (temperature of polymerisation = 50°C) and SKS-50 PKH (temperature of polymerisation = 100°C) by using fatty acids as emulsifiers, and also latex mixtures for manufacturing foam rubber (car seats) etc. It was found that the plasticity of the latex plays an important part during the manufacture of the foam, and that it also affects the properties of the foam itself. Methods and conditions for manufacturing the special latex SKS-50 were investigated by the Leningrad Branch of the Research Institute for the Tyre Industry (NIIKP) (Nauchno-issledovatel'skiy institut kabel'noy promyshlennosti (NIIKP)) and the Factory "Sevkabel", as well as conditions for coating the cables with a thin layer of insulation. The authors explained the influence of the plasticity of the polymer contained in the latex on the quality of the rubber goods,

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SOV/138-58-4-2/13

On the Influence of Plasticity of Rubber on the Technological Properties of Synthetic Latexes.

and modified the methods of production by taking their hypothesis into account. Investigations are carried out at present, in conjunction with the NII Tyre Industry, on the influence of the plasticity of the polymer on its adhesive properties. The plasticity of a polymer also influences the process of manufacture of rubber goods by ion - precipitation. In particular, in chloroprene latex the molecular characteristics of a polymer influence considerably the rate of ageing of these latexes and the quality of the rubber articles. The influence of the plasticity of the rubber contained in the latex on the use and properties of manufactured goods requires further investigation. Laboratories should evolve more standards and more universal methods of defining the plasticity of the polymer in the latex. There are 5 References: 1 English and 4 Soviet.

ASSOCIATION: All-Union Research Institute for Synthetic Rubber im. Academician S. V. Lebedev (Vsesoyuznyy nauchno-issledovatel'skiy institut sinteticheskogo kauchuka im. akad. S. V. Lebedeva).

Card 3/3

1. Synthetic rubber--Properties 2. Rubber--Plasticity

SOV/138-58-5-2/9

AUTHORS: Lebedev, A.V.,
~~Fernor, N.A.~~
Mints, S.M.,
Zakharchenko, P.I.,

TITLE: The Vulcanisation of Synthetic Latexes (Vulkanizatsiya sinteticheskikh lateksov)

PERIODICAL: Kauchuk i Rezina, 1958, ¹Nr 5, pp 3-9 (USSR)

ABSTRACT: The method and conditions for the sulphur-vulcanisation of some synthetic latexes were investigated, as well as the characteristics of laminae prepared from the same. The latexes were vulcanised by intensive gamma radiation without using chemical vulcanisation agents. It was found that the strength of laminae made from these latexes depends on the polarity of the rubber. The latexes tested included 1,3-butadiene-styrene rubbers SKS-30A, SKS-50PG And also the 1,3-butadiene-nitrile rubber SKN-40; the composition and conditions of polymerisation are given in Table 1. Experiments on radiation vulcanisation were carried out in the Physico-Chemical Institute im. L.Ya.Karpov by using ⁶⁰Co as a source of radiation with an activity of

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SOV/138/58-5-2/9

The Vulcanisation of Synthetic Latexes

600, 1400 and 20,000 Curie. The samples were placed in 15 - 35 ml glass ampuls and subjected to radiation (intensity = 0.14 - 1.3 mil.roentgen units/hour). No coagulation of the latex could be observed. Laminae were prepared from the vulcanised latex by using aqueous fixing agents. Comparative tests were carried out under identical conditions with laminae prepared from latexes containing vulcanisation agents. The physical and mechanical characteristics of these laminae were determined. The degree of vulcanisation was evaluated as well as their solubility. Benzene was used as solvent and swelling agent for 1,3-butadiene-styrene rubbers and for "Revul'teks" (Revultex obtained from Revertex Limited) and acetone for 1,3-butadiene-nitrile rubber. The laminae were treated with methyl alcohol, saturated with phenyl-B-naphthylamine to extract the admixtures. The physical and mechanical properties of the laminae are listed in Table 2 and for comparative properties

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The Vulcanisation of Synthetic Latexes

data is included on vulcanised laminae made of non-vulcanised latexes and also of a sample of "Revul'teks". The mechanism of formation of laminae from vulcanised latexes was investigated: the strength of dry laminae from latexes vulcanised with sulphur appears to be the result of the cumulative action of Van der Waals forces and of chemical bonds between the globules. Table 4: data on the strength of the laminae (in%) at varying degrees of humidity. Changes in the physical and mechanical properties and the solubility of the laminae from vulcanised latexes after 8 - 10 months of storing were investigated; results of these investigations (Tables 5 and 6) show that the ageing of polymers in latexes proceeds with greater intensity than in the laminae when Vulkatsit-R-extra-N is used. The properties of the laminae made from latexes vulcanised when applying varying amounts of gamma

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The Vulcanisation of Synthetic Latexes

radiation (Tables 7 and 8) are discussed. There are 8 tables, 1 figure and 23 references of which 15 are English, 1 French, 1 German, 1 Japanese, 1 Dutch and 4 Soviet.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy institut sinteti cheskogo kauchuka im.S.V.Lebedeva (The All-Union Scientific-Research Institute for Synthetic Rubber imeni. S.V. Lebedev)

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SOV/138-58-7-1/19

AUTHORS: Lebedev, A.V., Fermor, N.A., Selivanovskiy, S.A., and Beresnev, V.N.

TITLE: Some Technical Properties of Chloroprene Latexes Depending on the Size of Particles and the Saturation of the Adsorption Coatings (Nekotoryye tekhnologicheskiye svoystva khloroprenovykh lateksov v zavisimosti ot velichiny chastits i nasyshchennosti adsorbtsionnykh obolochek)

PERIODICAL: Kauchuk i rezina, 1958, ¹Nr 7, pp 1 - 5 (USSR)

ABSTRACT: The rate of ionic deposition, the rate of syneresis in water, the rate of drying and setting of coatings and physico-mechanical properties of the gel of chloroprene latexes having particles of various sizes, were investigated. To some latex samples soap was added in order to compare the properties of latexes: a) at an equal degree of saturation of the globules of the coating and b) at an identical weight ratio of the emulsifier to the polymer. Polymerisation was carried out in a 50-litre apparatus at 25 - 30 °C (Table 1). Initiators and emulsifiers usually used during the synthesis of chloroprene latexes were used (Refs 14 and 15). The size of the particles and the degree of saturation was determined by

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SOV/138-58-7-1/19

Some Technical Properties of Chloroprene Latexes Depending on the
Size of Particles and the Saturation of the Adsorption Coatings

adsorption titration of the latexes with solutions of sodium oleate and resin soap (Refs 9 and 10). The physico-mechanical properties of the raw gel were defined with a Kublanov dynamometer (Ref 12) and the physico-mechanical properties of dry vulcanised coatings with a Shopper dynamometer according to the VNIISK methods (Ref 11). Heat ageing of the latexes was effected in an air thermostat for 36 hours at 70 °C. An analysis of data given in Table 2 and Figures 1-3 shows that the rate of ionic deposition in the initial period (within the limits of experimental error) is equal for all tested samples; in the following period it is higher for latexes with large particles. The weight ratio of the raw and dry gel for all samples and in all stages of ionic deposition remains approximately constant (about 2.2). The average rate of ionic deposition increases with increasing degree of saturation of the globules with emulsifiers. If the latex contains very small particles and the globules are less saturated with emulsifiers, syneresis of the gel proceeds more quickly and more completely in the aqueous

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SOV/138-58-7-1/19

Some Technical Properties of Chloroprene Latexes Depending on the Size of Particles and the Saturation of the Adsorption Coatings

medium (Table 3). From simple calculations, it can be established that within the limits of investigated sizes of particles and of degree of saturation, the rate of syneresis and its extent are approximately proportional to the specific exposed surface of the polymer particles; the proportional coefficient is considerably higher for latexes stabilised with rosin soaps. When infra-red irradiation is applied the rate of drying of latex coatings is higher if large-particle latexes are used. However, the rate of separation of moisture decreases with increasing degree of saturation of the adsorption layers with emulsifiers. The amount of deposits and the reduction coefficient increase slightly during drying when the sizes of the particles and the degree of saturation of the adsorption layers increase. The specific elongation of gels from large-particle latexes is in all cases lower than the corresponding values for highly dispersed latexes. It decreases with increasing degree of saturation of the adsorption layers with the polymer globules.

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The physico-mechanical values of vulcanised layers decrease

SOV/138-58-7-1/19

Some Technical Properties of Chloroprene Latexes Depending on the Size of Particles and the Saturation of the Adsorption Coatings

with increasing soap content in the polymers; at equal soap content they do not (within the limits of experimental error) depend on the sizes of the particles in the latex. The raw gel, as well as the vulcanised layers from latexes, stabilised with sodium resinate, have better physico-mechanical properties than the corresponding gels and coatings stabilised with sodium oleate. This is due to the different solubilities of calcium salts of rosin and oleic acids in chloroprene. There are 3 figures, 3 tables and 15 references, 5 of which are Soviet, 7 English and 3 German.

ASSOCIATION: Vsesoyuznyy nauchno-issledovatel'skiy institut sinteticheskogo kauchuka im. S.V. Lebedeva
(All-Union Research Institute for Synthetic Rubber im. S.V. Lebedev)

Card4/4

1. Chloroprenes--Polymerization 2. Chloroprenes--Physical properties 3. Chloroprenes--Mechanical properties 4. Chloroprenes--Test results 5. Synthetic rubber--Preparation

FERMOR, N.A.; PRYZNER, A.B.

Using synthetic latices as a base in preparing paints. Bnl.
tekh.-ekon.inform. no.5:20-23 '59. (MIRA 12:8)
(latex) (Paint)

PEYZNER, A.B.; FERMOR, N.A.

Making foam-rubber articles of synthetic latex. Biul.tekh.-
ekon.inform. no.8:15-17 '59. (MIRA 13:1)
(Rubber goods)

S/138/59/000/012/003/006

AUTHORS: Peyzner, A. B., Uzina, R. V., Fernald, N. A., Khazanovich, I. G.

TITLE: The Basic Factors Determining the Type of Divinyl-Styrene
Latex in Tire Cord Impregnation

PERIODICAL: Kauchuk i Rezina, 1959, No. 12, pp. 10-14

TEXT: The effect of the emulsifier, the polymerization depth, the polymerization temperature, the plasticity of the polymer, the ratio of divinyl and styrene in the polymerizing mixture on the strength of the bond of the rubber-cord system in the polymerization of divinyl-styrene latexes was studied. 1) The emulsifier: It was found that by replacing Nekal with soaps of paraffinic acids and hydrated colophony the strength of the bond system is increased significantly. This is explained by an increase in the physico-mechanical properties of the adhesive film and by the intensity of the intermolecular interaction at the adhesive-rubber interface (Ref. 7) (Table 2). In switching over to low-temperature polymerization latexes the stated advantages are retained. 2) The polymerization depth: The bond strength of the impregnated cord decreases in the case of divinyl-styrene latexes of low-temperature polymerization at a conversion depth of 60%.

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The Basic Factors Determining the Type of Divinyl-Styrene Latex in Tire Cord Impregnation

3) The polymerization temperature: It was found that the highest bond stability was obtained at a temperature of 20°C (Fig. 3). This is explained by the combination of good elastic properties of the polymer at low-temperature polymerization and a certain branching of the chains observed in a switch-over from 5 to 20°C. At 20°C the polymerization takes place more rapidly, the 60% conversion depth is reached after 9 hours instead of 24 hours (Fig. 4). 4) The polymer plasticity: With an increase in the polymer plasticity the bond stability of the system and the physico-mechanical properties of the adhesive film pass through their optimum value at a plasticity of 0.15-0.25 according to Karrer (1,500-2,800 g hardness according to Defoe) (Fig. 6). This is explained by the fact that the high plasticity of the polymer ensures favorable conditions for molecule diffusion from the adhesive into the rubber lining, but does not ensure the necessary mechanical properties of the adhesive (Refs. 4, 9). A decrease in the plasticity of the polymer raises the mechanical properties of the adhesive and thus limits the mobility of molecules and lowers their diffusibility which leads to a decrease in the bond strength. A polymer with an average plasticity (0.15-0.25 according to Karrer) ensures the best adhesion. ✓

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The Basic Factors Determining the Type of Divinyl-Styrene Latex in Tire Cord Impregnation

5) The styrene ring content: The presence of styrene in the divinyl-styrene latex (over 30 weight parts) does not increase the strength of the adhesive-rubber system. Fig. 7 shows that an increase in the styrene content in the polymerizing hydrocarbon mixture increases the physico-mechanical properties of the film adhesive; the stability of the bond between the impregnated cord and the rubbers reaches its optimum value at 30 weight parts of styrene. At a styrene content of more than 30 weight parts the adhesive hardness increases. This brings about unfavorable conditions for the diffusion of the adhesive polymer rings and for the polymer compatibility. 8) Selection of the divinyl-styrene latex type for the impregnation of the tire cord: As a result of the investigations conducted a specific latex is recommended for this purpose. The CKC-30ШХП (SKS-30ShKhP) divinyl-styrene latex was obtained according to given specifications and was found to surpass the CKC-30Ш (SKS-30Sh) latex both in the bond strength between the impregnated cord and the rubbers and by the physico-mechanical properties of the adhesive film (Table 3). The authors point out that by using the recommended latex the strength of the bond between the individual parts of the tire, the durability of the tires in stationary tests and the roadability of the

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S/138/59/000/012/003/006

The Basic Factors Determining the Type of the Divinyl-Styrene Latex in Tire Cord Impregnation

tire during performance is improved. There are 3 tables, 8 graphs, 10 references: 7 Soviet and 3 English.

ASSOCIATION: Vsesoyuznyy Nauchno-issledovatel'skiy Institut sinteticheskogo kauchuka im. S.V. Lebedeva i Nauchno-issledovatel'skiy institut shinnoy promyshlennosti (The All-Union Scientific Research Institute of Synthetic Rubber im. Lebedev and Scientific Research Institute of the Tire Industry) ✓

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